

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

Tsuyoshi OKAMOTO et al.

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Examiner: VASISTH, VISHAL V

Title : LUBRICANT FOR WATER-MISCIBLE METAL WORKING OIL

DECLARATION UNDER RULE 132

Honorable Commissioner of Patents and Trademarks,  
Alexandria, Virginia 22313-1450

Sir:

I, Tsuyoshi Okamoto, a citizen of Japan and having postal mailing address 14-12, Tsukudacho, Nishikyogoku, Ukyo-ku, Kyoto 605-0813 JAPAN, declare and say that:

In March 1994, I graduated from Graduate School of Engineering, Osaka University, and received a master's degree in Chemistry;

From April 1994, up to the present, I have been employed by Sanyo Chemical Industries, Ltd., and engaged in the works of research and development for Surfactant;

In April 1997, I joined the group where the inventors of the present invention belonged;

I am the inventor of the above-identified application and am familiar with the technical field of the present invention;

I have read the Official Action mailed and the references cited therein. I respectfully submit herewith my exact report;

In order to demonstrate that KOH cannot be used as a catalyst for a THF/AO random addition, and that a polyether compound including a branched oxybutylene group is inferior in lubricity to a polyether compound including a linear oxybutylene group, I have carried out the following experiments.

According to Examples in the specification of the present invention, polyethers were produced using alcohol, alkylene oxide and catalyst. In addition, water-based metal working oils containing obtained polyethers were obtained in the composition ratio corresponding to polyether (E2). Then, water dilution stability and lubricity of these water-based metal working oils were evaluated.

<Polyether (E'3)>

90 parts (1 mol) of 1,4-butanediol, 1,008 parts (14 mol) of THF and 5.0 parts of KOH were charged into the same autoclave as Example 1, and thereafter 880 parts (20 mol) of EO was dropped thereinto through the pressure-resistant dropping funnel over 10 hours while maintained at a temperature of 35 to 50°C. Thereafter, the solution was aged at a temperature of 50°C for 5 hours and thereafter cooled. Then, in order to confirm polymerization, about 10 g of content was sampled out of the autoclave. 2 g of the sample was weighed precisely and putted into a circulation-type drier at a temperature of 105 °C for 2 hours. The dry weight was 0.1 g (unreacted 1,4-butanediol).

Therefore, it is concluded that the polymerization of THF was not substantially found at all, and that polyether (E'3) was not obtained.

For reference, in the case of Example 2, the dry

weight was 1.9 g, and the polymerization ratio was 95 %.

<Polyether (E'4)>

90 parts (1 mol) of 1,4-butanediol and 5.0 parts of KOH were charged into the same autoclave as Example 1, and dehydrated under a reduced pressure. Thereafter, 1,008 parts (14 mol) of 1,2-butylene oxide (BO) and 880 parts (20 mol) of EO were dropped thereinto through the pressure-resistant dropping funnel over 10 hours at a temperature of 105°C. Then, the solution was aged over 5 hours until the pressure reached equilibrium at a temperature of 130°C and then cooled, and refined in the same manner as Example 1 to obtain 1,850 parts of a 14 mol-BO and 20 mol-EO random adduct of 1,4-butanediol (E'4), which was regarded as a lubricant.

<Polyether (E'5)>

92 parts (1 mol) of glycerin and 8.0 parts of KOH were charged into the same autoclave as Example 1, and dehydrated under a reduced pressure. Thereafter, 1,152 parts (16 mol) of BO and 1,760 parts (40 mol) of EO were dropped thereinto through the pressure-resistant dropping funnel over 10 hours at a temperature of 105°C. Then, the solution was aged over 5 hours until the pressure reached equilibrium at a temperature of 130°C and then cooled, and refined in the same manner as Example 1 to obtain 2,800 parts of a 16 mol-BO and 40 mol-EO random adduct of glycerin (E'5), which was regarded as a lubricant.

Results

The yield, Mw, HLB and cloud point of polyether (E2), (E5), (E'3), (E'4) and (E'5) are shown in Table 1. The results of evaluation of water dilution stability and

lubricity of water-based metal working oils containing each polyether are also shown in Table 1.

Table 1

Production Name of Polyether			(E2)	(E5)	(E'3)	(E'4)	(E'5)
Conditions of Production	Random AO Addition Reaction	1,4-Butanediol	90 (1)		90 (1)	90 (1)	
		Glycerin		92 (1)			92 (1)
		Propylene Glycol					
		THF	1008 (14)	1152 (16)	1008 (14)		
		1,2-Butylene oxide				1008 (14)	1152 (16)
		EO	880 (20)	1760 (40)	880 (20)	880 (20)	1760 (40)
		BF <sub>3</sub> •THF	12.8	19.1			
		KOH (catalyst)			5.0	5.0	8.0
	Yield (part)		1,880	2,850	No polymerization	1,850	2,800
	Mw		2,150	3,300		2,150	3,300
Property	HLB		10	12		11	13
	Cloud point (°C)		21	39		22	42
	Appearance of Diluent with Water		Excellent	Excellent		Excellent	Excellent
Lubricity	Lubricity to Steel Plate	Oil Film Shortage	Excellent	Excellent	No data	Poor	Poor
		Friction Coefficient	0.093	0.095		0.175	0.182
	Lubricity to Aluminum Plate	Oil Film Shortage	Excellent	Excellent		Poor	Poor
		Friction Coefficient	0.073	0.075		0.173	0.179

### Conclusion

From the results shown above, the following facts 1

and 2 can be clearly read.

1. When KOH is used as a catalyst for a THF/AO random addition, the polymerization of THF is not substantially found at all, and polyether (E'3) is not obtained.

Therefore, it is concluded that KOH cannot be used as a catalyst for a THF/AO random addition

2. Water dilution stability and lubricity of water-based metal working oils containing polyether (E'4) and (E'5) are inferior to those of water-based metal working oils containing polyether (E2) and (E5).

Therefore, it is concluded that the polyether compound including the branched oxybutylene group is inferior in lubricity to the polyether compound including the linear oxybutylene group.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed this                      day of      *June 30*      , 2009

*Tsuyoshi Okamoto*

Tsuyoshi Okamoto